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13. ABSTRACT (Maximum 200 words)

Over the course of this 3-year program, dramatic improvements have been made to the MOCVD process. In the first year of the program, we began to explore the effect of increasing film thickness on J_c of YBCO tapes fabricated by MOCVD. In the second year, we expanded this study, and obtained more data on this effect over a wide range of film thickness. 5 microstructural causes were determined for reduction in I_c with increasing film thickness:

secondary phases, roughness, a-axis grains, texture, and cap/buffer layer uniformity. Also, we initiated work on developing processing techniques to address some of these microstructural causes. One such work was smoothing of the rough surfaces in thick films using Gas Cluster Ion Beam (GCIB) smoothing. GCIB smoothing was shown to partially remove this dead layer, decrease surface roughness by 24% to 42% and increase I_c up to 86%. In the third and last year of the program, we worked on addressing the microstructural causes for reduction in I_c in thick films. We have studied alternate superconductor composition that results in less film roughness and less interfacial reaction with the buffer layer. We have also developed high-rate MOCVD process (without any assist means such as photo-assist) to minimize film exposure to high temperature for long time periods. Microstructural analysis has been conducted on films deposited at different deposition rates. Finally, we demonstrated high critical currents on meter-long moving tapes produced by the MOCVD process using high deposition rates

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**Final Report to AFOSR
Contract # F49620-00C-0021
September 2003**

by

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Objectives :

The objective of the program is to develop a fundamental understanding of YBCO film deposition by Metal Organic Chemical Vapor Deposition (MOCVD) on biaxially-textured metal substrates. In comparison to other vapor deposition techniques, MOCVD offers the advantages of rapid deposition rates, film uniformity over large areas, and is not limited to line-of-sight deposition. The program includes a study of the

- a) influence of MOCVD processing conditions such as the flow rate of precursor vapors, precursor vaporization temperatures, oxygen partial pressure, reactor pressure, and the deposition temperature on the film features such as superconducting phase formation, composition, texture, deposition rates, uniformity in thickness, porosity and the presence of secondary phases,
- b) relationship between film microstructure and the critical current density (J_c), and
- c) influence of metal substrate and buffer layers on the growth and performance of YBCO

Status of Effort :

Over the course of this 3-year program, dramatic improvements have been made to the MOCVD process. In the first year of the program, we began to explore the effect of increasing film thickness on J_c of YBCO tapes fabricated by MOCVD. In the second year, we expanded this study, and obtained more data on this effect over a wide range of film thickness. 5 microstructural causes were determined for reduction in J_c with increasing film thickness: secondary phases, roughness, a-axis grains, texture, and cap/buffer layer uniformity. Also, we initiated work on developing processing techniques to address some of these microstructural causes. One such work was smoothing of the rough surfaces in thick films using Gas Cluster Ion Beam (GCIB) smoothing. GCIB smoothing was shown to partially remove this dead layer, decrease surface roughness by 24% to 42% and increase J_c up to 86%. In the third and last year of the program, we worked on addressing the microstructural causes for reduction in J_c in thick films. We have studied alternate superconductor composition that results in less film roughness and less interfacial reaction with the buffer layer. We have also developed high-rate MOCVD process (without any assist means such as photo-assist) to minimize film exposure to high

temperature for long time periods. Microstructural analysis has been conducted on films deposited at different deposition rates. Finally, we demonstrated high critical currents on meter-long moving tapes produced by the MOCVD process using high deposition rates.

Experimental :

MOCVD of YBCO was conducted in a custom-built facility at SuperPower described in previous Progress reports. The surface morphology of the YBCO films was examined by Field Emission Scanning Electron Microscope (FESEM) followed by compositional analysis by Energy Dispersive X-ray Spectroscopy (EDS). Film cross sections were made with Focussed Ion Beam Milling (FIB). The texture of the films was analyzed by XRD including polefigure measurements. The thickness of the films was measured by surface profilometry.

Accomplishments/New Findings :

1. Alternate superconductor composition :

Up to this point, YBCO is the material of choice for coated conductors. However, it may not be the best choice for thick film coated conductors. We began evaluation of alternate rare-earths for thick film coated conductors. One candidate is samarium. We have effectively developed a MOCVD process for SmBCO coated conductors. Our results show that the microstructure of SmBCO is considerably smoother than that of YBCO as shown in **Figure 1**.

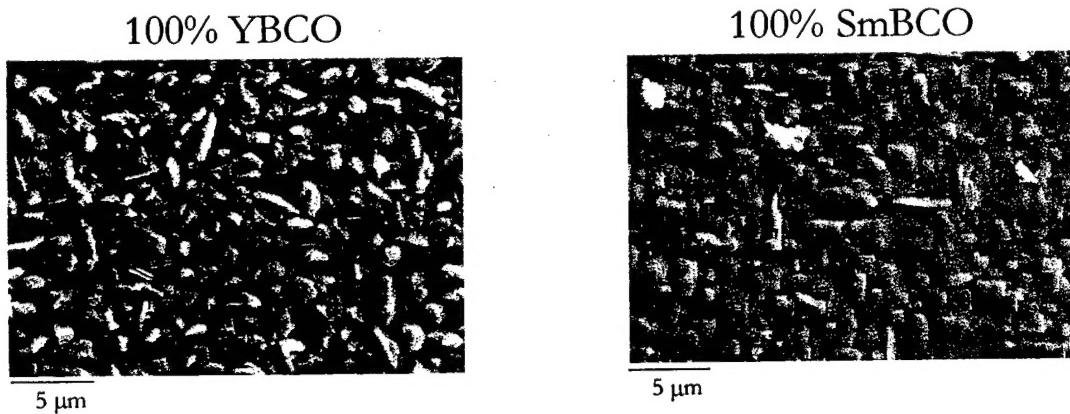


Figure 1. Surface microstructures of YBCO and SmBCO coated conductors prepared by MOCVD

To determine the reason for the better surface smoothness with SmBCO compared with YBCO, we etched out the HTS film layer and examined the microstructure of the underlying buffer. Results of the microstructure of the buffer layer is shown in **Figure 2**. The microstructure of the buffer layer in the etched SmBCO film is considerably smoother than that of the buffer layer in the etched YBCO film. The reason for this difference is believed to be less interfacial reaction between the cap layer and SmBCO compared to YBCO. Less interfacial reaction between the cap layer and SmBCO could lead to a better template for epitaxial film growth.

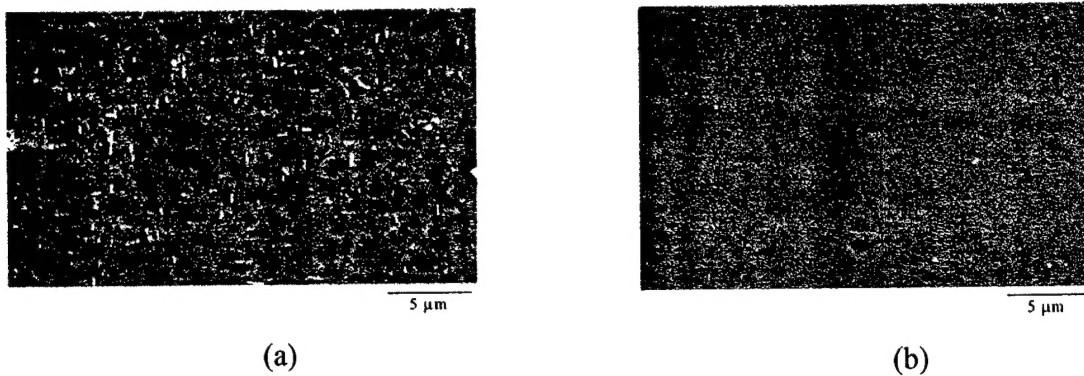


Figure 2. Surface microstructures of buffer layer after etching out the YBCO (a) and SmBCO (b) layers

Earlier work on SmBCO films indicated that its J_c is substantially lower than the J_c of YBCO films. In fact, that was one of the reasons provided by LANL for investigating multilayering of YBCO and SmBCO. We have evaluated the entire range of Sm substitution of Y in YBCO coated conductors and the J_c results are shown in **Figure 3**. The performance of the coated conductor is relatively constant irrespective of the amount of substitution of Sm. We also found optimization of 100% SmBCO films can result in a performance of 100 A, as shown in figure. This result could very well be the first demonstration of 100 A in a SmBCO coated conductor.

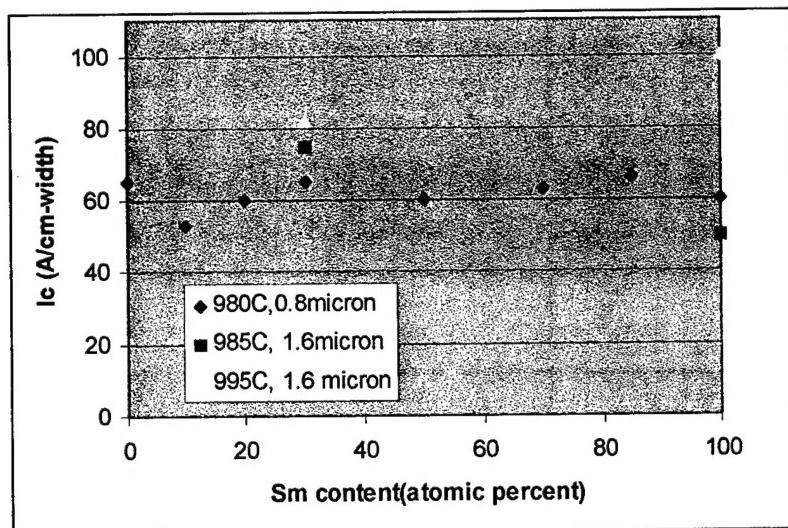


Figure 3. Effect of Sm doping on the critical current of YBCO conductors. The effect of temperature (heater temperatures shown) on the critical current of 30% SmBCO and 100% SmBCO is also included.

Another advantage of using SmBCO is its higher T_c . Results from transition temperature measurements obtained over a range of Sm doping of YBCO are shown in **Figure 4**. It can be seen that 100% SmBCO exhibits about 3 K higher T_c than 100% YBCO.

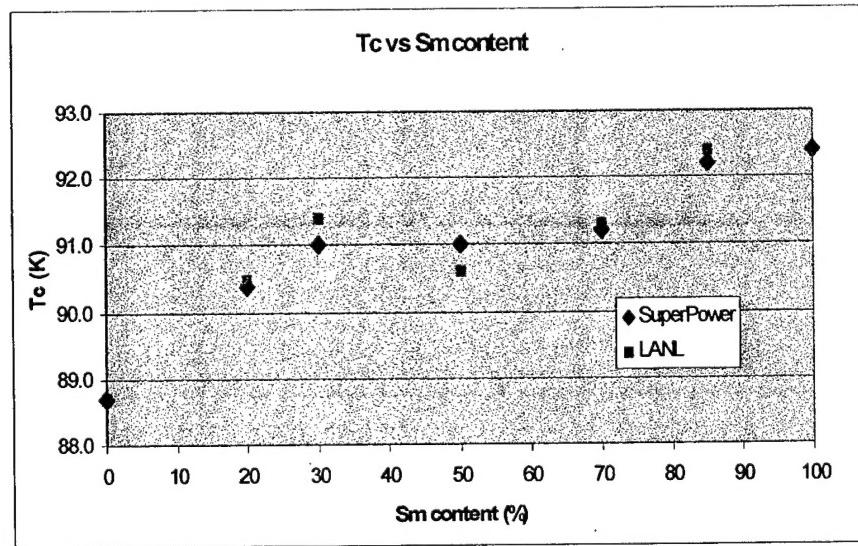


Figure 4. Effect of Sm doping on T_c of SmBCO conductor prepared by MOCVD. Measurements were made at both SuperPower and LANL as indicated in the figure.

The improved surface smoothness obtained with SmBCO is attractive for thick film fabrication. Thick films of a single composition would be easier to scale up compared to multilayers.

2. Higher deposition rates :

When depositing thick films, the tape has to be exposed to high temperatures for long time periods. When depositing films of a few microns in thickness, initial deposited layers could degrade as they are exposed to high temperatures for long time periods. Using an elegant etching process, Los Alamos National Laboratory demonstrated evidence of such a degradation of underlying layers when depositing 5 micron thick films. To avoid such degradation, it would be important to limit the time of exposure to high temperatures which would be possible if higher deposition rates are achieved. One approach is to increase the rate of precursor delivery. In our studies with 1 – 1.5 micron thick films, we have been able to achieve deposition rates as high as 120 Angstroms/second just by increasing the precursor delivery rate. This result is shown in **Figure 5**. The figure also shows that there is further room for increasing the deposition rate by additional increase in the precursor flow rate.

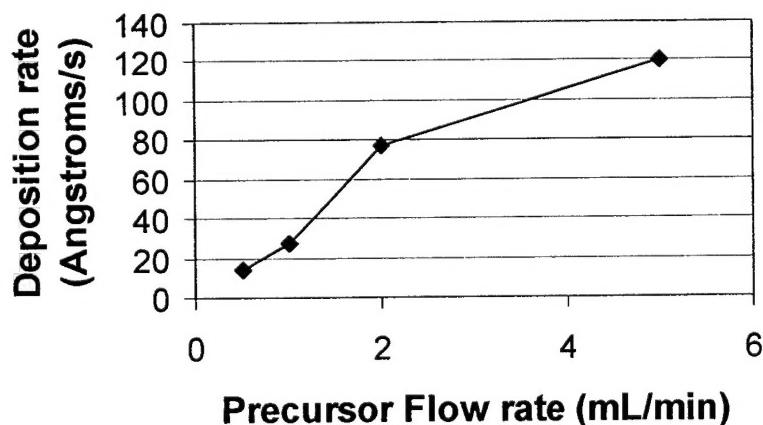


Figure 5. Demonstration of high deposition rates in our MOCVD process by increase in precursor flow rates.

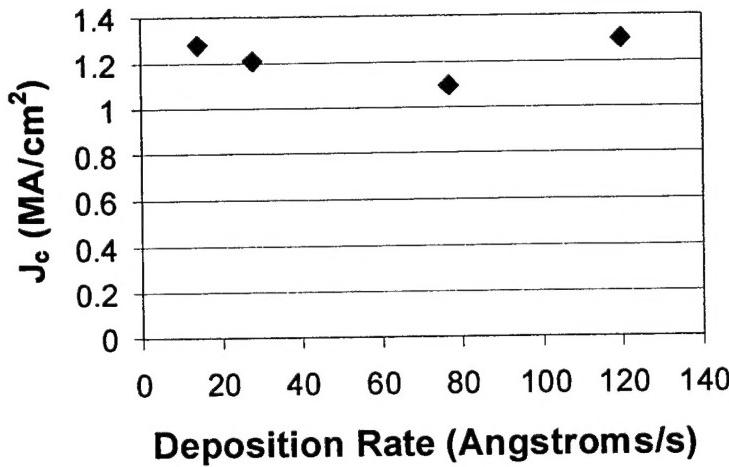


Figure 6. Achievement of high critical current densities in MOCVD-based coated conductors even at high deposition rates.

The rate of 120 Angstroms/second is quite high considering no assist mechanisms such as photo-assist was used. Not only we achieved such high deposition rates, but also demonstrated J_c greater than 1 MA/cm² at these rates. Figure 6 shows the critical current densities of coated conductors fabricated by MOCVD at various deposition rates. It can be seen from the figure that a J_c greater than 1 MA/cm² was achieved at all rates studied so far. The J_c of the conductor prepared at 120 Angstroms/s is higher than the trend up to 80 Angstroms/s since we spent more time on optimizing the MOCVD process at that rate.

This is more evident in the next figure.

It is not only important to achieve 1 MA/cm² performance, but for coated conductors, it is even more critical to demonstrate critical currents greater than 100 A/cm-width. Figure 7 exhibits the critical current performance of MOCVD-based coated conductors prepared at various tape speeds. The first 4 data points correspond to the data shown in figure 6. A deposition rate of 120 Angstroms/second was used at tape speeds of 7.5, 10, and 15 m/h. We have demonstrated critical currents greater than 100 A/cm-width even at a tape speed of 7.5 m/h. The trend in figure 7 shows decreasing critical current with tape speed primarily because of reduced film thickness at the higher tape speeds. The I_c of the conductor prepared at 7.5 m/h at 120 Angstroms/s shows a higher I_c value of 150 A/cm-width since we spent more time on optimizing the MOCVD process at this tape speed. We

believe that higher values of critical current can be achieved even at 10 and 15 m/h after further process optimization.

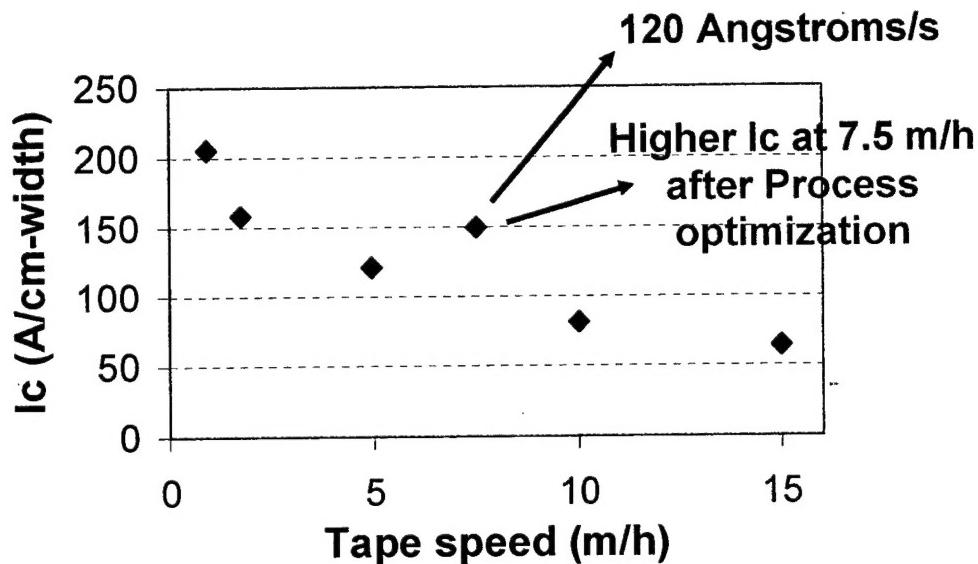


Figure 7 Critical current of MOCVD-based coated conductors fabricated at various tape speeds (deposition rates)

The process used to achieve high values of critical current at the high deposition rate has been transferred to longer tape lengths. Figure 8 shows the critical current performance (current-voltage characteristic) of a meter-long MOCVD tape fabricated with a deposition rate of 120 Angstroms/s using a tape speed of 5 m/h. A critical current of 193 A was achieved over the meter-long tape.

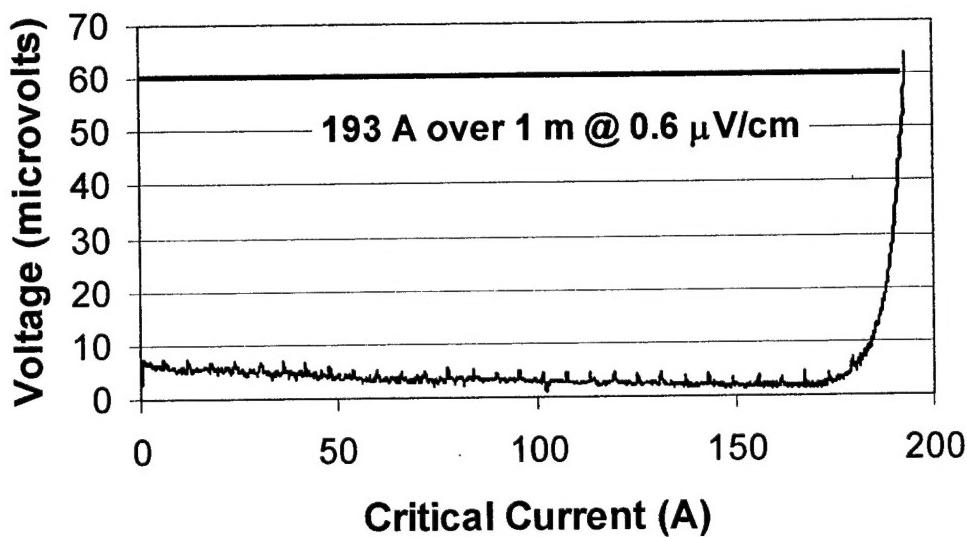


Figure 8 Critical current of a meter-long MOCVD-based coated conductors fabricated at a tape speed of 5 m/h and a deposition rate of 120 Angstroms/s.

Personnel Supported :

Dr. Hee Gyoun Lee, Sr. Materials Scientist

Dr. Jodi Reeves, Sr. Materials Scientist

Publications :

1. V. Selvamanickam, H. G. Lee, Y. Xie, X. Xiong, Y. Qiao, Y. Li, J. Reeves, and A. Knoll, "High Performance Coated Conductors using MOCVD", Proc. Coated Conductor for Applications Workshop, Orta, September 2003.
2. V. Selvamanickam, H. G. Lee, Y. Li, X. Xiong, Y. Qiao, J. Reeves, Y. Xie, A. Knoll, and K. Lensedt, "Fabrication of 100 A Class, 1 m long Coated Conductor Tapes by Metal Organic Chemical Vapor Deposition and Pulsed Laser Deposition", Proc. ISS, Yokohama, November 2002, Physica C. (in print)

Interactions/Transitions :

a. Conference Presentations :

1. MRS Fall meeting, Boston, December 2002
2. DOE Coated Conductor Workshop, St. Petersburg, January 2003
3. American Ceramic Society Meeting, Nashville, April 2003
4. Coated Conductor for Applications Workshop, Orta, September 2003
5. EUCAS, Sorrento, September 2003

b. Interaction :

SuperPower has been collaborating AFRL-WPAFB in the development of YBCO coated conductors. Samples of MOCVD tapes have been provided to AFRL for various measurements. We have collaborated with the U. Kansas on studying the thickness dependence of J_c by ion milling experiments. SuperPower has augmented the AFOSR-funded program through CRADAs with Argonne National Lab (ANL) and Los Alamos National Lab (LANL). ANL and LANL have been assisting in the characterization of the YBCO tapes. SEM, TEM, AFM, and FIB analysis have been conducted by SuperPower staff and students supported by SuperPower at U. Albany.

c. Transition :

The ongoing AFOSR program will have a large impact on ongoing materials and device development programs at SuperPower. *The AFOSR program is a critical program at SuperPower for the development of YBCO coated conductors.* The success of the program will lead to a high-performance and potentially lower cost replacement for Bi-2223 conductor. Bi-2223 conductor is currently the main HTS conductor available in long lengths for all device programs at Intermagnetics such as transformers, cables, fault-current controllers, and generators. Based on its superior performance and potential lower cost, YBCO is the clear choice for HTS conductor for all these devices. *SuperPower recognizes this fact and is strongly supporting the AFOSR program through funds for capital equipment including the MOCVD facility.* Last year, SuperPower committed to over \$ 1 M of its funds towards capital equipment for MOCVD facilities that include a new Pilot MOCVD facility, a MOCVD research system, and modifications to the existing Prototype MOCVD facility. The AFOSR program will eventually enable the fabrication of a high

performance superconducting tape that can find wide use in military, electric power, magnetic, medical and applications.

8. Inventions :

None.

9. Honors & Awards :

None

10. Research Planned for Following Year :

This is the end of the 3 year program. We proposed and have been awarded a new 3-year program to continue the work on MOCVD-based coated conductors. In that program, we will specifically work on achieving high J_c in thick films as well as improve the magnetic field dependence of J_c of coated conductors fabricated by MOCVD.